Bureau of Standards, (Div. VIII)
Department of Commerce
Washington, D. C.



Letter Circular 104
October 15, 1918.

# HEAT TREATMENT OF STEEL.

The modern practice of the heat treatment of steel has become so complex, involving as it does a very definite knowledge of the thermal changes occurring in the metal as it is heated or cooled, i.e. the critical transformations; the initial microstructure of the metal and the structural changes which may be effected by various treatments; correct methods of heating and cooling; the modifications of practice induced by the introduction of various alloying elements, etc., that even an elementary discussion of the subject becomes involved and complex. Below are listed a few of the salient points in the various fields of the heat treatment of steel, a thorough knowledge of which is absolutely essential for the proper handling of material in these operations.

Steel is essentially an alloy of iron and carbon (disregarding for the moment alloys which are added to impart certain properties). The carbon is present as a definite compound with a certain amount of the iron, i.e. $Fe_3C$, called cementite; it is the condition of this cementite which governs the physical properties of the steel.

In the stable state, at ordinary temperatures, this cementite is separated (precipitated) from the ferrite (as the iron is called) and can be recognized under the microscope as a separate structural constituent. If the steel is heated, the carbide passes into solution in a certain amount of the iron at a temperature of about 725° Cent., represented by the line PK, Fig. 1. As the temperature is raised, this solution gradually absorbs the remaining ferrite, until the respective temperature or the line GOS is reached (which depends on the carbon content), when the whole will be a single solid solution. (This discussion refers to steels of less than 0.9% carbon. Above that percentage, the excess constituent is cementite instead of ferrite. Hence the range PS to GS represents absorption of ferrite, the range SK to SE represents absorption of cementite. At 0.9% all the dissolving occurs at once.) Steel in such condition preserved down to room temperature would show but one constituent under the microscope. The solid solution which is preserved by very rapid cooling is much harder at room temperature than the separated condition which is readily obtained by annealing. Intermediate states are obtained by varying the speed of cooling from the high temperatures and also by slight re-heating of a rapidly cooled specimen. The temperature at which the carbide passes into solution may be detected with a pyrometer, because of the absorption of heat when the change takes place. The temperature range in which this change takes place is called the critical range of the steel.

The physical properties of the steel can often be judged from the "grain", which in works practice is seen in the fracture, and in the laboratory may be seen in the microstructure. In general, coarse grain is taken to indicate weakness and brittleness, while fine grain would indicate strength and toughness. Coarse grain is produced by long heating at high temperatures, and by passing through the critical range slowly in cooling. Fine grain is found in samples heated to but slightly above the critical range and cooled more or less rapidly. Very coarse grain may be reduced by forging, and by cold work followed by annealing.

Any of the various heat treatments applied to steel may be reduced to a combination of the following elements; (1) heating at a definite temperature for a definite length of time, (2) cooling from this temperature at some desired rate, and (3) re-heating if desired. The more complex treatments consist in the duplication of one or more of these elements together with changes in the various temperatures, rates of cooling, etc. These elements will be considered under the designations -- annealing, hardening and tempering.

## I. ANNEALING.

### a. Purpose.

1. To soften for machining.

2. To give certain physical properties, e.g. to soften it for some definite service, or make it stronger and less brittle by refining the grain.

3. To remove stresses due to cold-working, quenching, etc.

### b. Temperature.

For a. (1) and a. (2), the temperatures chosen are usually the same, and may be found in the shaded area in Fig. 1. It will be seen that they vary with the carbon content of the steel; they are further modified, sometimes to a marked degree, by alloys present. For softening in machining it is not absolutely necessary to heat above the critical range.

For a. (3), there is in the case of cold-worked material a fairly definite temperature at which the effects of cold work are removed. This usually lies between 500° and 575° Cent.; that is, somewhere in that range re-crystallization takes place and the elongated grains divide and become equi-axed. Re-heating after quenching will be considered under "Tempering".

c. Rate of heating.

The size and the shape of the piece must be considered, it being especially important in large and irregular pieces not to force the heating, as the outside of the article and thin parts would be greatly overheated, Pieces should be arranged in the furnace to insure as even heating as possible, and in gas furnaces should be set on supports to allow free passage of the gases underneath the piece.

d. Rate of cooling.

This is determined, in the case of annealing above the critical range, by the properties desired, by the size of the piece and by the carbon content. For maximum softness the piece is allowed to cool in the furnace. Very large pieces may be removed from the furnace and cooled in the air, their size preventing too rapid cooling. On the other hand, thin pieces of high carbon content cool rapidly enough in air to become quite hard.

"Normalizing" may be considered under the head of annealing. It consists in heating the steel above the critical range and cooling in air, and serves to put the steel in an undressed condition of good crystal size and arrangement. It also minimizes the danger of quenching cracks in high-carbon steels, especially on repeated hardening.

e. Double Annealing.

For maximum grain refining. Heating to a temperature somewhat above the critical range, then air cooling or quenching, followed by reheating to a temperature just below the critical range.

f. Divorce Annealing.

Annealing to spheroidize the cementite. Long heating at just below the critical temperature converts the carbide from plate form to granular form; makes steel especially tough.

g. Protection during heating.

Neutral or reducing ("hazy") flames when burning gas or oil. Heating in lime or carbon. Annealing boxes.

### h. Overheating.

Too high annealing temperature; too long heating (size of piece must be considered). "Burning" of steel, i.e. heating to region of incipient fusion. Overheated steel can be restored by suitable heat treatment, especially in connection with forging. Burnt steel cannot be restored.

## II. HARDENING.

### a. Temperature.

Must be above the critical range; piece should be heated long enough to insure complete solution (special precautions for alloy steels); too high temperature must be avoided. The annealing range shown in Fig. 1 may be taken as a guide for quenching temperatures. The higher the carbon, the greater the precautions to keep the quenching temperature but little above the critical range. In steels having more than 1% carbon, normalizing from above the upper critical range (line SE, Fig. 1) may precede quenching, if excess carbide is in plate form which would weaken steel.

### b. Method of heating.

1. Open furnace. Precautions same as in annealing.

2. Baths. Molten lead, barium chloride, sodium chloride, etc. Advantages of accurate temperature control, uniform heating, no danger of overheating. Salt baths give clean steel on quenching, but decarburize the surface with long heating.

### c. Quenching.

1. Baths. Different liquids have different speeds of cooling and so impart different degrees of hardness to steel on quenching. The following represents an average of the data from many different sources, and indicates roughly the relative hardening that may be expected.

| Quenching Medium. | Time of cooling specimen from 800° C to 250° C. |
|---|---|
| Water at 20°C | 1.0 |
| " " 50°C | 1.2 |
| " " 80°C | 1.4 |
| " " 100°C | 3.0 |

| | |
|---|---|
| Brine at 20°C | 1.0 |
| " " 60°C | 1.0 |
| " " 80°C | 1.2 |
| Alcohol 20°C | 1.0 |
| Corn Oil 20-75°C | 1.3 |
| Fish Oil | 1.5 to 1.9 |
| Lard Oil | 1.6 to 1.8 |
| Sperm Oil | 1.7 |
| Linseed Oil | 1.8 |
| Cottonseed Oil | 1.9 |
| Mineral Oils 20°C | 1.3 to 2.5 |
| Mercury 20°C | 2.5 |
| Glycerine 20°C | 3.0 |
| Air 20°C | 15. |

2. Temperature of cooling bath. Quenching power decreases as temperature rises, but oil changes very little. Salt solutions do not change as much as water. Cooling coils for cooling the bath. Amount of quenching liquid should be sufficient in relation to size of piece to prevent undue rise of temperature of bath. Stirring the bath and moving the piece to cause contact with cool liquid.

3. Precautions. To prevent warping, thin pieces should be quenched axially, should not have excessive scale, should be normalized after cold-working before quenching.

d. Double Quenching.

## III. TEMPERING.

(a) Purpose. Relieves stresses due to quenching, reduces brittleness (especially in water-quenched pieces), toughens the steel. Elasticity for springs.

(b) Temperatures. Any temperature below the critical range, but usually between 100° and 500° Cent.

100°-150°C: Razors and other hard cutters, (brittleness not objectionable).

200°-300°C: Cutting tools (chisels, milling cutters, knives, etc.)

300°-400°C: Springs. High elasticity.

Above 400°C: Great toughness and resistance to shock.

(c) Regulation of Tempering.

1. Time at tempering temperature. Longer heating causes greater softening. Long heating at low temperatures better than short heating at high temperatures (gives greater uniformity).

2. Rate of cooling of little influence, as there is no transformation. Pieces often quenched for convenience.

3. Temper color scale (see Fig. 2b). Color due to formation of thin surface layer of oxide. True indication of degree of softness at surface (when there is free access of air): longer heating at same temperature gives darker color.

4. Tempering baths: oil, fused salts, fused metals. Advantages: accurate temperature control, uniformity.

5. "Blazing off" (heating to flash point of oil after oil quench). Undesirable: uneven heating, excessive heat at surface.

6. Relation of tempering temperature to the severity of the quench.

7. Removal of large pieces from the quenching bath while they are still hot enough to temper of themselves. Exact regulation difficult.

## IV. CASE HARDENING.

(a) Purpose: generally, to impart a very hard wearing surface with a softer and more resilient interior for withstanding shock, as in cams, gears, etc.

(b) Composition of base steel: usually about 0.2% carbon; some alloys advantageous, especially a small amount of chromium.

(c) Carburizing. Article heated for suitable length of time in solids or molten baths.

Solids: mixtures of charcoal, charred leather, bone, etc., with barium carbonate or other carbonates. 5 to 20 hours at 875°-950°C. Deep case.

Liquids: molten potassium cyanide, sometimes mixed with chlorides, other cyanides, etc. 1 to 15 minutes at 800°-900°C. Thin case. (Highly poisonous).

"Pack Hardening". Heating just above critical range (750°C.) while packed in solid cements, about two hours. Quench in oil. Used on high-carbon steels.

Protection of portions to be left uncarburized,-plating with copper, etc.

(d) Heat treatment: the aim is to produce a hard case, with an interior refined of the effects of overheating and rendered capable of resisting shock. After carburizing with

(1) solids,- double quench first from above critical range of core, to refine grain of core after the long heating, then from just above critical range of case, to harden the case <u>properly</u>.

(2) liquids,-carburizing bath usually kept at hardening temperature and piece quenched directly.

Temper as desired.

## V. ALLOY STEELS.

Though the heat treatment of such steels is, in principle, essentially like that of the plain carbon steels, the introduction of the alloying element, e.g. nickel, chromium, tungsten, etc. modifies the practice so profoundly that each type requires a special study.

In general, the addition of the special element may modify the treatment and therefore the properties in the following ways:

(a) By the lowering of the critical ranges or transformation temperatures, forms which in plain carbon steels are stable only at high temperatures may become the stable forms even upon slow cooling. Nickel is one illustration of this behavior.

(b) The critical changes may be made to take place much more slowly than in carbon steels of the same carbon content so that the transformation temperature is raised upon heating and lowered upon cooling. Chromium illustrates this behavior. In general, the alloy steels are much slower in their response to heating than are the plain carbon steels, thereby necessitating a longer heating period or a higher temperature without exposing the steel to the danger of becoming so coarse as would be the case if the special element were not present.

(c) The structural condition in which the special element exists determines largely the properties of the steel and to a large extent the heat treatment necessary to develop the requirement properties, i.e. whether the added element enters into the ferrite constituent (nickel is a case of this), or into combination with the

carbon (as with chromium and tungsten). The double carbide formed in the latter cases increases in mineral hardness of the steel without increasing the brittleness to such a degree as an addition of an equivalent amount of carbon would do.

(d) By the addition of two or more special elements, the advantages of each may be retained and the disadvantages largely neutralized, as in chrome-nickel steels which as a type probably represent the best "all-around" alloy steels in commercial use for general purposes.

References to some of the books on the subject of special steels must be had for definite information upon the special alloy steels their heat treatment, characteristic properties, and special uses for which each is adapted.

Most of the steel companies manufacturing special grades of steel have worked out, experimentally, the heat treatment necessary to bring out the most desirable physical properties of their particular steel; upon purchasing, directions are given concerning the heat treatment.

## VI. HIGH SPEED STEEL.

The introduction of large amounts of alloying elements in high speed steels caused radical changes in the nature of the steel. The heat treatment therefore differs greatly from that usually applied to steels. Hardening is effected in principle as before, by bringing the carbides into solution in the iron and cooling at such a rate as will leave them in solution. The presence of large amounts of tungsten and chromium changes both the temperature required for effecting the solution and the allowable range of cooling rates.

Annealing. This is effected by heating at 850°-900°C. for periods sometimes as long as 5 hours, depending on the nature of the steel.

Hardening. The steel is heated very slowly to about 700°C., then very rapidly to about 1200°C. and cooled in any way desired, air, air-blast, oil or water. The rate of cooling has no great influence on the hardness, as the carbides remain in solution except on extremely slow cooling.

Tempering. High speed tools are generally not tempered, although tempering at 500°-600°C, is recommended for some tools, After water-quenching, it may be advisable to remove cooling stresses by tempering at a low temperature.

Special Alloys. Certain alloys, not properly steels, have come into use as high speed cutters. "Stellite", a cobalt-chromium-tungsten alloy, requires no heat treatment and responds to none. It is cast to shape and ground. Other unusual combinations of chromium, nickel, zirconium, molybdenum, etc. have been used, the nature of the alloy determining whether it requires heat treatment.

The following list includes many of the most recent and helpful books on the subject of heat treatment.

Steel and Its Heat Treatment, D.K. Bullens, (1916) - (1918)
John Wiley & Sons, publ's.

The author has explained and correlated the theoretical and practical aspects of the general subject in such a way as to make the book one of the most valuable (from the standpoint of the practice) in English.

Metallography and Heat Treatment of Iron and Steel, Albert Sauveur (1916), Pub. Sauveur & Boylston, Cambridge, Mass.

An excellent discussion of the microstructure of steel and the structural changes induced by heat treatment is given, without entering into the "practical" aspects of the subject.

Heat Treatment of Steel, E. Oberg (1917)
The Industrial Press, New York.

Practical directions for heat treatment and furnace practice.

Composition and Heat Treatment of Steel, E.F. Lake, (1911)
Publ. McGraw-Hill Co., New York.

General account of the metallurgy and treatment of steel.

Iron and Steel (A Pocket Encyclopedia), H.P. Tiemann
McGraw-Hill Book Co., New York, (1910)

Very helpful for anyone concerned with the manufacture or use of steel and iron.

Hardening, Tempering, Annealing and Forging of Steel, Jos. V. Woodworth, (1911) New edition 1916.
N. W. Henley Pub. Co., 132 Nassau St., New York City.
Practical to the extreme. Detailed directions are given for the heat treatment of all kinds of tools, etc.

Steel: Its Selection, Annealing, Hardening and Tempering.
E.R. Markham, (1913)
N.W. Henley Pub. Co., 132 Nassau St., New York City.

This is of the same general nature as the above reference. It should prove of value to one already somewhat familiar with the general nature and behavior of steel.

The Heat Treatment of Tool Steel, H. Brearley, (1911)
Longmans, Green & Co., New York.

A clear exposition of the proper method of treatment of high carbon steel is given.

Hardening and Tempering Steel, Cassell & Co., New York City (1911)

A workshop guide to the heat treatment of steels including high speed steels.

American Machinists Handbook, F. H. Calvin and F. A. Stanley (1909)
McGraw-Hill Co., New York City.

Subjects other than heat treatment are also considered, necessarily the discussions are rather brief and concise.

Americam Soc. Test. Matls. Year Book, Philadelphia, Pa.

The society has adopted as standard practice the recommendations of Committee A-4 on annealing of carbon steel rolled and forged pieces and castings, also for the heat treatment of case hardened carbon steels. See A 35-11, A 36-14, A 37-14.

Trans. of Soc. of Automotive Engineers, 1790 Broadway, New York.

The American Society of Automotive Engineers through its Iron and Steel Division has issued some very definite instructions for the heat treatment of practically all the various carbon and alloy steels which enter into motor car constructions.

The following books are also valuable:

G. Mars, Die Spezialstahle, ihre Geschichte, Eigenschaften, und Herstellung. (1912)

Leon Guillet, Les Aciers Speciaux.

Jean Escard, Les Metaux Speciaux. 1909.

Bradley Stoughton, Metallurgy of Iron and Steel,
McGraw-Hill Co. 1916

F. W. Harbord, The Metallurgy of Steel, chap. 39,
Griffin's Met. Series.

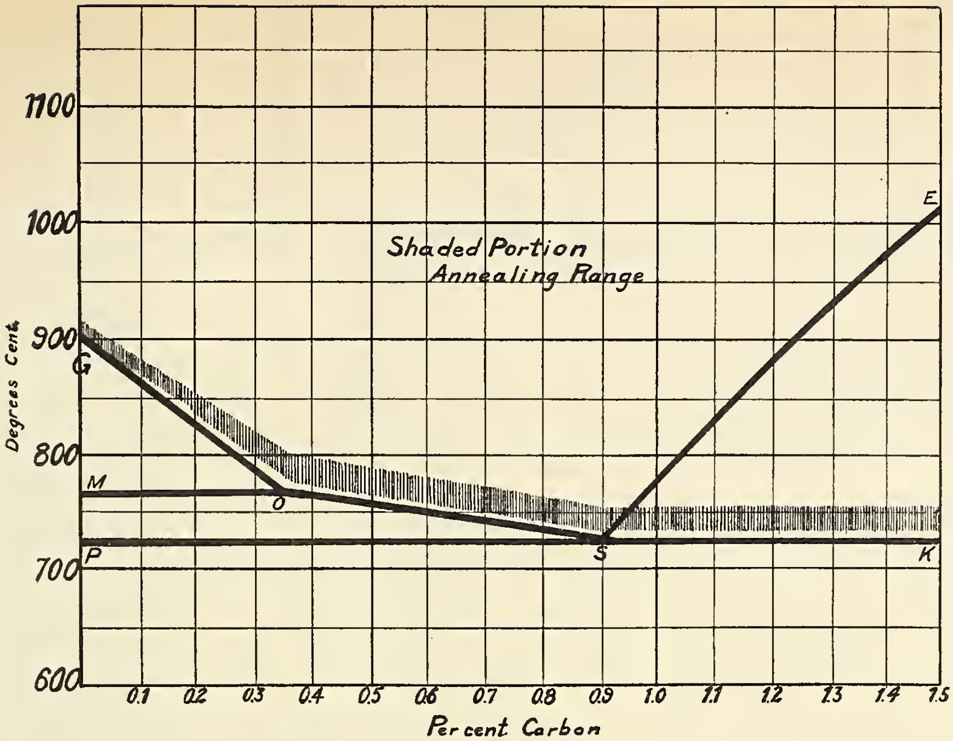
Hannemann, H., Metallographie und Wärmbehandlung; particularly valuable from the microstructural viewpoint.

C. A. Edwards, The Physico-Chemical Properties of Steel. J. B. Lippincott & Co., Philadelphia, 1916.

H. Brearley, Case Hardening of Steel, 1914. Lliffe & Sons, London, publrs.

Giolitti, F.: The Cementation of Steel, Trans. from Italian By Richards and Rouiller, McGraw-Hill & Co., 1915.

W. Rosenhain, Introduction to Physical Metallurgy, (1915), Chapter XII.




- Fig. 1

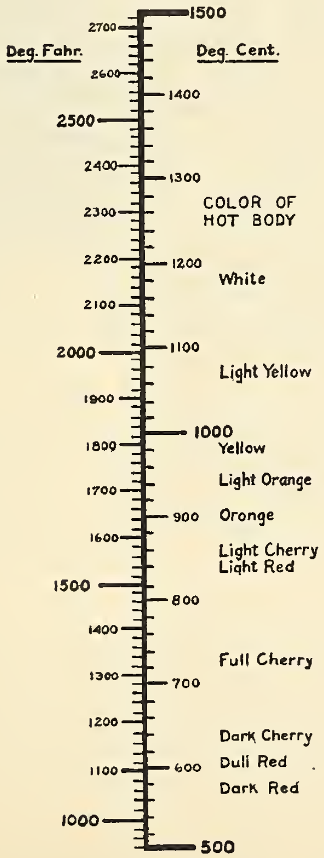



Fig. 2a

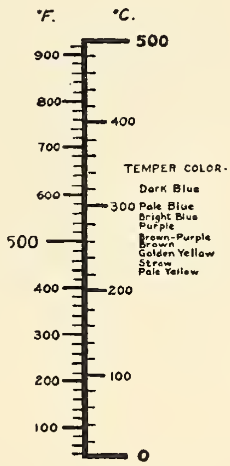



Fig 2b